

R75-17  
14 NOV 1974

Reprinted from TEXTILE RESEARCH JOURNAL, Vol. 44, No. 11, November 1974  
Printed in U. S. A.

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## Sorptive Textile Systems Containing Activated Carbon Fibers

GILBERT N. ARONS, RICHARD N. MACNAIR, LAURANCE G. COFFIN, AND HUBERTINA D. HOGAN

*U. S. Army Natick Laboratories, Natick, Massachusetts 01760, U. S. A.*

### ABSTRACT

Activated carbon fabrics and yarns were shown to effectively sorb gases, vapors, and dissolved solids. Fabrics prepared by Arons and Macnair (phenolic precursor) and Maggs (viscose precursor) were the most activated of all the materials studied. The yarns had a lower activity but probably can be improved to have comparably high sorption capacity. Other materials studied included permeable textile systems made by mechanically reinforcing carbon fabrics and yarns with stronger materials. For example, selected fabrics were laminated or quilted between two other fabric layers while the yarns were knitted or woven with other yarns.

### Background

Sorbent textile forms of activated carbon fibers are now becoming available for use in specific applications, and a new technology is developing from the production and utilization of these textiles. A recently developed commercial use is air filters in automobile air conditioners. Other applications are presently in the

research and development stage. These could include: protective clothing for manufacturing and laboratory uses; air filters in respirators, clean rooms, and room air conditioners; and structures for chemical processing to aid in the purification of solutes and solvents.

This paper is concerned with the development of material for use in protective garments which would

afford protection against hazardous chemical vapors. Powdered forms of activated carbon presently impregnated in clothing are not entirely satisfactory. Carbon particles must be physically entrapped in polymeric binders to remain in the material. This is detrimental to sorption and must be compensated for by incorporating an excess of carbon into the system to obtain a desired sorption level. Furthermore, impregnation ultimately produces clothing having reduced air permeability and increased weight. These factors in addition to thickness, the major factor, contribute to excessive physiological stress experienced by the wearer when protective garments are worn in a hot environment.

Activated carbon fibers are now being incorporated in experimental textile structures in attempts to circumvent the above problems. Fibrous forms of carbon can be supported in textile systems by other stronger fibers and yet be completely exposed to the sorbate, thus offering high sorption efficiency. Sorbent textiles should have relatively high air permeability combined with reduced weight and thickness and offer a considerably reduced heat stress level.

### Introduction

Commercial carbon fibers are generally prepared from man-made precursor fibers such as PAN, viscose, or pitch. In the past decade considerable fundamental information was obtained on the carbonization of these and other textile precursors. As a result, carbon (and graphite) textiles in the form of fiber, yarn, and fabric have been adopted for use in composites, especially for aerospace applications [13].

The introduction of oxidation techniques resulted in the preparation of highly activated carbon fibers. Such materials are characterized by high surface areas, in the order of 250 to 1200 m<sup>2</sup>/g, resulting from small pore formation. Pores may be classified according to size as follows: micropores having less than 30 Å diameter, transitional pores in the 30 Å to 2000 Å diameter range, and macropores over 2000 Å diameter.

Recently, Arons, Macnair, and Erickson [3, 4] reviewed briefly the state of the art for activated carbon textiles and described some products made from fibrous phenolic and viscose precursors. In addition, Adams, Boucher, Cooper, and Everett [1, 2, 7] have conducted intensive investigations on carbon fibers made from Saran.<sup>1</sup> This material is a classical precursor which is used in fundamental studies because the chemistry of its decomposition is relatively simple. Bailey and Maggs [5] were issued a British patent describing the production of activated carbon fabric and other fibrous forms from viscose precursors treated with Lewis acids.

<sup>1</sup> Saran is a product of the Dow Chemical Co., Midland, Michigan. Citation of this and subsequent trade names does not constitute an official endorsement by the U. S. Army Natick Laboratories.

Bolwell [6] reported on the vapor sorption capacity of this British carbon fabric. Economy and Lin [9] were granted a German patent covering the production of high surface-area carbon fibers from a nonmelttable, hardened, phenol-formaldehyde (novolak) precursor. Two companies, Lonza Ltd., and F. J. Burrus & Cie [12], share a British patent which describes hollow, activated carbon fibers.

Another way to produce sorbent fibers containing activated carbon is largely of academic interest—namely, to spin fibers from polymer loaded with activated carbon. McDowell [14] described viscose monofilaments containing 80% activated carbon, and Smisek [15] referred to attempts to incorporate active carbon into synthetic fibers. Recently, Buisson [8] was granted a German patent on active-carbon-filled polyamide(imide) fibers.

This paper describes the characterization of selected, activated carbon yarns and fabrics which have potential as sorbents for gas, vapor, and liquid systems. Included are some unique textile systems which provide mechanical reinforcement of activated carbon yarns and fabrics to overcome deficiencies of low strength, elongation, and abrasion resistance [10, 11]. The characterization consisted of a series of tests which are common in different technologies. The series included textile tests, activated carbon characterization tests, sorption tests, and physiological comfort-index tests. Physical properties normally obtained on textile materials were measured on all of the carbon yarns, fabrics, and their related fabric systems. Conventional activated carbon analyses for surface area, pore diameter, and pore volume were obtained on the all-carbon materials. Static vapor sorption tests were run on all-carbon yarns and fabrics. Dynamic vapor sorption tests were run on selected fabrics which were either all-carbon or systems containing carbon. Liquid phase sorption capacity was determined on one all-carbon fabric previously described [4]. A thermal transfer test relevant to clothing comfort in climatic extremes was performed on representative samples of each fabric system containing carbon.

### Experimental

**MATERIALS.** Two types of materials were studied: all-carbon and reinforced carbon materials. All-carbon fabric and yarn samples are described in Table I. The nine fabrics (A-I) contained staple yarns, and the two yarns (J, K) contained continuous filaments. These materials were made by carbonizing and activating the corresponding viscose and phenolic (Kynol<sup>2</sup>) precursors.

Reinforced carbon fabrics (shown in Table II) were constructed from the two carbon yarns described above and various stronger yarns of other types. The

<sup>2</sup> Kynol is a product of the Carborundum Company, Niagara Falls, New York.

TABLE I. Carbon fabrics and yarns.

Carbon Material	Precursor <sup>a</sup>	Source
Fabric A	Viscose	3M Company
Fabric B	Viscose	F. A. P. Maggs <sup>b</sup>
Fabric C	Viscose	F. A. P. Maggs <sup>b</sup>
Fabric D	Viscose	F. A. P. Maggs <sup>b</sup>
Fabric E	Phenolic	Carborundum Company
Fabric F	Phenolic	G. N. Arons & R. N. Macnair <sup>c</sup>
Fabric G	Phenolic	G. N. Arons & R. N. Macnair <sup>c</sup>
Fabric H	Phenolic	G. N. Arons & R. N. Macnair <sup>c</sup>
Fabric I	Phenolic	G. N. Arons & R. N. Macnair <sup>c</sup>
Yarn J	Viscose	Union Carbide Corp.
Yarn K	Viscose	3M Company

<sup>a</sup> Precursor form was the same as the final form after carbonization.

<sup>b</sup> Chemical Defence Establishment, Porton Down Salisbury Wilts, England.

<sup>c</sup> Prepared at U. S. Army Natick Laboratories as a single batch of four pieces.

TABLE II. Reinforced fabrics constructed with carbon yarns.

- Fabric L—Weft insertion warp knit made with Carbon Yarn J filling reinforced with Nomex yarn (Fig. 1).
- Fabric M—Woven twill made with polyester braid covered Carbon Yarn J filling reinforced with Nomex warp (Fig. 2).
- Fabric N—Woven twill made with Carbon Yarn J and cotton yarn (Yarn No. 30/2) in the filling (3 carbon and 2 cotton repeating) reinforced with Nomex warp (Fig. 3).
- Fabric O—Woven twill made with Carbon Yarn K filling reinforced with Nomex warp (Fig. 4).
- Fabric P—Woven satin composite made with Carbon Yarn J filling yarns exposed on both face and back; reinforcing Nomex warp yarns exposed on face only; and reinforcing cotton warp yarns (Yarn No. 50/2) exposed on back only (Fig. 5 and 6).

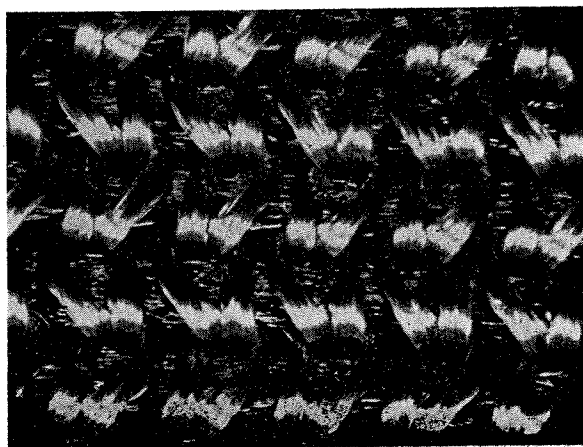


FIG. 1. 44X Magnification of Fabric L (wale side).

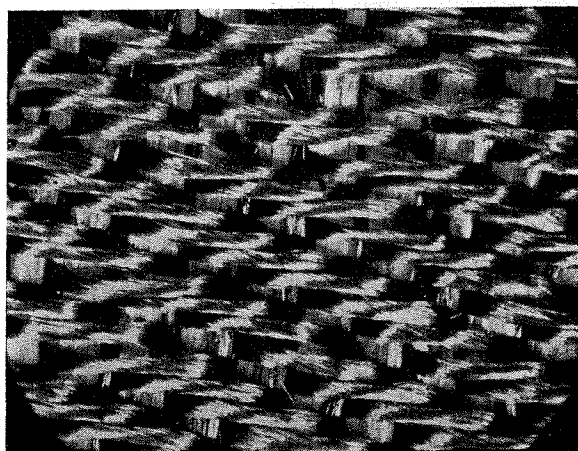


FIG. 2. 22X Magnification of Fabric M.

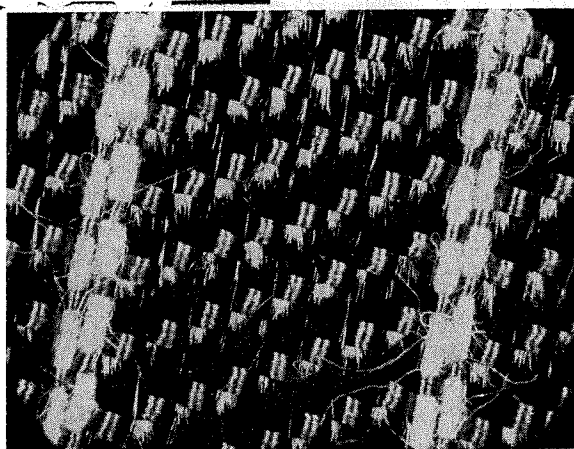


FIG. 3. 22X Magnification of Fabric N.

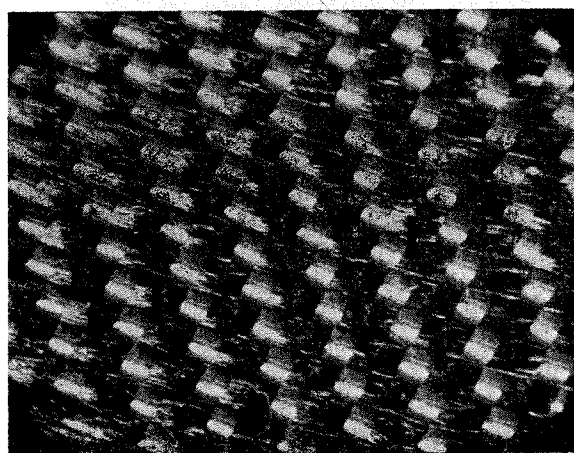


FIG. 4. 22X Magnification of Fabric O.

materials shown were selected after a series of initial trials conducted to achieve satisfactory samples having a reasonably high carbon yarn content, *i.e.*, in the range of 6 to 9 oz/yd<sup>2</sup>. In all cases, 200-den Nomex<sup>3</sup> aramid

<sup>3</sup> Product of the duPont Company, Wilmington, Delaware.



FIG. 5. 22X Magnification of Fabric P (Nomex face side).

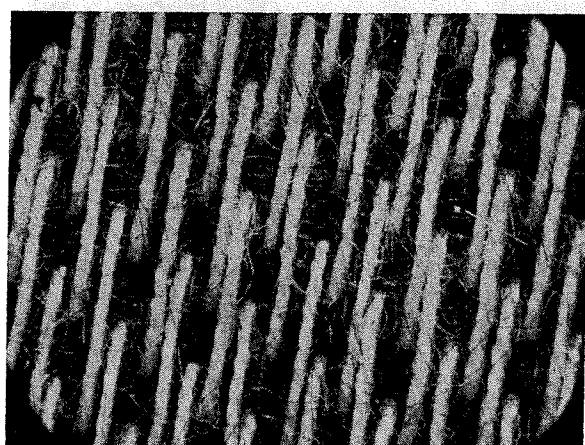


FIG. 6. 22X Magnification of Fabric P (cotton backing side).

continuous filament yarn was used as the supporting structure. Nomex aramid was selected because of its durability and high temperature resistance. Filament yarn was selected because of relatively low cost and ready availability. Spun yarn may ultimately prove to be more practical but was not originally considered because initial procurements in the yarn number and twist level desired would require relatively large quantities of fiber. In addition to the Nomex, 16 carriers of 70-den polyester yarn were braided over a Carbon Yarn J core in one sample. Other samples incorporated cotton yarns both for mechanical support and protection of the carbon yarns. Figures 1 through 6 are low-power photomicrographs of these samples.

In another type of reinforcement material Carbon Fabric A (Table I) was laminated between 5 oz/yd<sup>2</sup> nylon/cotton twill and 2 oz/yd<sup>2</sup> nylon tricot. Each of three laminates contained a different thermoplastic adhesive fabric (2 layers) which was activated in a heated garment press. These are described in Table III.

TABLE III. Laminates of Carbon Fabric A with nylon-cotton twill and nylon tricot.<sup>a</sup>

Sample	Adhesive Fabric	
	Description	Source
Laminate Q	Thermogrip LM5230A, Polyamide, 1.15 oz/yd <sup>2</sup>	USM Chemical Co.
Laminate R	Thermogrip 5030A, Polyester, 1.23 oz/yd <sup>2</sup>	USM Chemical Co.
Laminate S	Delnet X-230, Polyolefin, 0.53 oz/yd <sup>2</sup>	Hercules, Inc.

<sup>a</sup> Lamination sequence as follows:

_____	Nylon-Cotton Twill
_____	Adhesive Fabric
_____	Carbon Fabric A
_____	Adhesive Fabric
_____	Nylon Tricot

Quilting was another technique used to reinforce a carbon fabric. Quilt T was prepared by stitching Carbon Fabric E (Table I) between two layers of plain weave Nomex fabric, each weighing 3.3 oz/yd<sup>2</sup>. The sample was prepared using a diamond pattern having 2-in. leg lengths.

In addition to the above, Witco Grade 718 granular activated carbon,<sup>4</sup> which has a 20 × 40 mesh granule size and is rated for 1250 m<sup>2</sup>/g surface area, was selected for comparison with the carbon fibers in liquid phase sorption experiments. In these experiments CI Solvent Red 24 in toluene, conc 2.000 g/l., was used to follow the progress of dye sorption from solution by both a fabric and the granular form of carbon.

**TESTS. Textile Tests.** Whenever feasible, textile tests performed on carbon yarns, carbon fabrics, and related systems were accomplished in accordance with Federal Test Method Std. No. 191 [16]. Table IV indicates the applicable Test Method No. used.

TABLE IV.—Textile test methods.<sup>a</sup>

Test	Method No.
Fabric thickness	5030.2
Fabric weight	5041
Fabric texture (yarns per inch)	5050
Fabric breaking strength and elongation	5100
Fabric burst strength	5120
Fabric air permeability	5450
Yarn denier	4021
Yarn breaking strength and elongation	4100

<sup>a</sup> Federal Test Method Standard No. 191.

**Activation Tests.** A static CCl<sub>4</sub> vapor sorption test was used as a screening tool to measure the approximate extent of activation of each carbon yarn and fabric. The sorption chamber consisted of a desiccator contain-

<sup>4</sup> Product of Witco Chemical Corp., New York, N. Y.

ing one liter of  $\text{CCl}_4$  and maintained at  $25^\circ\text{C}$ . Carbon samples in weighing bottles were allowed to sorb the saturated solvent vapor until they reached a maximum constant weight. The percent weight increase was then calculated.

Activation was then examined in greater detail as the carbon yarns and fabric were tested for surface area, pore diameter, and pore volume. The macropore and transitional pore size distributions for each sample were measured by the use of an Aminco<sup>5</sup> 60,000 psi Mercury-Intrusion Porosimeter. The density for each sample was determined from the mercury intrusion data and corrected for the total pore volume. The pore size distribution and surface area for each sample were calculated from sorption and desorption isotherm data obtained on an Aminco Adsorptomat, using nitrogen as the sorbate after each sample had been degassed at  $70^\circ\text{F}$ .

**Sorption Testing.** Sorption of hazardous vapors was determined for selected fabric samples (all carbon or systems containing carbon) in dynamic tests which do not approach equilibrium conditions.<sup>6</sup> In the basic procedure the vapor is passed through a  $100\text{-cm}^2$  fabric sample at a selected concentration,  $90^\circ\text{F} \pm 0.5^\circ$ ,  $80\% \text{ RH} \pm 2\%$ , and with a face velocity of  $10 \text{ cm/min}$  ( $1 \text{ liter/min}$  flow rate). The effluent stream is monitored by passage into a specific solvent and the amount of absorbed vapor subsequently estimated colorimetrically with a developer system. Details are summarized in Table V.

Liquid phase sorption capacity of Pluton B-1 carbon fabric for an aqueous dye solution was previously studied using Methylene Blue (U.S.P.) [4]. In the

present work the sorption of another dye, CI Solvent Red 24, from an organic (toluene) solution was studied using the same procedure. Freundlich sorption isotherms of the carbons were plotted for comparison with similar data for the blue dye.

**Physiological Stress Tests.** Thermal transmittance properties of fabric systems were evaluated on a guarded hot plate between the heated surface and a cool atmosphere. These data were translated into physiological stress data relevant to clothing comfort in climatic extremes. The procedure followed was that specified in ASTM Test Method D1518-64.

## Results and Discussion

The all-carbon fabrics and yarns described previously in Table I have textile properties which are indicated in Tables VI and VII, respectively. Typical cotton materials are shown for comparative purposes. In general, the carbon fabrics are in the  $5$  to  $7 \text{ oz/yd}^2$  weight range with a fairly uniform thickness of  $0.020$  to  $0.023 \text{ in}$ . Air permeability, texture, and breaking strength vary widely depending upon specific fabric construction. The two carbon yarns were similar to each other except for the higher strength of Yarn K. However, both yarns had sufficient strength to enable them to be knit or woven into fabrics on commercial textile equipment.

Sorption and penetration characteristics of the all-carbon fabrics and yarns are shown in Table VIII. The open pore volume data (nitrogen adsorption and mercury intrusion) are characteristic of the two probes used. For the nitrogen molecule probe which has a  $4 \text{ \AA}$  diameter, few transitional pores appeared in the partial range of  $30 \text{ \AA}$  to  $300 \text{ \AA}$  diameter. Accordingly, the nitrogen pore volume represents micropores in the  $4 \text{ \AA}$  to  $30 \text{ \AA}$  diameter range. For the mercury intrusion probe up to  $60,000 \text{ psi}$  there was a lower limit of  $30 \text{ \AA}$  pore diameter. Actually, no transitional pores appeared in the complete range of  $30 \text{ \AA}$  to  $2000 \text{ \AA}$  diameter.

<sup>5</sup> American Instrument Company, Silver Springs, Maryland.

<sup>6</sup> The apparatus and procedure were developed by Paul B. Dawson, Department of the Army, Edgewood Arsenal, Maryland. The method can be adapted for use with any vapor to demonstrate sorbent performance of permeable protective clothing materials under certain practical environmental conditions where hazardous vapors may be present.

TABLE V.—Dynamic test conditions for hazardous vapors.

Vapor	Vapor Conc., $\mu\text{g/l}$ .	Specific Solvent	Developer
HD $(\text{ClCH}_2\text{CH}_2)_2\text{S}$	$25 \pm 2$	diethylphthalate	p-nitrophenyl-3-pyridine and piperidine
GB $\begin{array}{c} \text{O} \\    \\ \text{CH}_3-\text{P}-\text{O}-\text{CH}(\text{CH}_3)_2 \\   \\ \text{F} \end{array}$	$25 \pm 2$	dibutyl phthalate	o-dianisidine
VX $\begin{array}{c} \text{O} \\    \\ \text{CH}_3-\text{P}-\text{S}-\text{C}_2\text{H}_4-\text{N}[\text{CH}(\text{CH}_3)_2]_2 \\   \\ \text{OC}_2\text{H}_5 \end{array}$	$15 \pm 2$	hexylene glycol	p-nitrophenyl-3-pyridine and piperidine

TABLE VI. Textile properties of all-carbon woven fabrics.

Designation	Weight, oz/yd <sup>2</sup>	Thickness, in.	Air Perm., ft <sup>3</sup> /min/ft <sup>2</sup>	Weave	Texture, yarns/in. W × F	Breaking Strength, lb/in.		Elongation, %	
						Warp	Fill	Warp	Fill
Fabric A	6.6	0.021	178	Plain	30 × 31	7.4	14.0	5	21
Fabric B	4.8	0.023	180	(singles yarns) Plain	36 × 33	3.0	2.9	—	—
Fabric C	5.1	0.021	212	(2-ply yarns) Plain	35 × 35	1.9	1.7	3	18
Fabric D	5.4	0.022	220	(2-ply yarns) Plain	35 × 32	6.3	3.4	5	23
Fabric E	5.3	0.020	151	(2-ply yarns) 3 × 3 basket	60 × 60	5.0	7.9	3	10
Fabric F <sup>a</sup>	5.4	0.021	54	(singles yarns) 2/1 twill	68 × 50	2.5	1.2	—	—
VEE 1716 <sup>b</sup>	7.5	0.025	47	(2-ply yarns) 5 harness sateen	85 × 50	160	123	—	—

<sup>a</sup> Representative of Carbon Fabrics F, G, H, and I, prepared as part of the same batch of four samples.

<sup>b</sup> A typical cotton sateen work clothing fabric shown for comparison purposes.

TABLE VII. Textile properties of carbon yarns.

	Carbon Yarn J	Carbon Yarn K	Cotton Yarn <sup>a</sup>
Diameter (mm)	0.86	0.86	0.27 <sup>b</sup>
Denier	1530	1290	532
Breaking strength (lbs)	4.4	5.9	1.9
Tenacity (g/d)	1.3	2.1	1.6
No. of plies (continuous filament)	2	2	1

<sup>a</sup> Typical 10/1 cc Cotton yarn shown for comparison purposes. Source: "Handbook of Textile Testing and Quality Control" by Elliot B. Grover and D. S. Hamby, N. Y., Interscience, 1966, p. 396.

<sup>b</sup> Estimated, based on yarn bulk density of 1.0 g/cm<sup>3</sup>.

Accordingly, the mercury pore volume represents macropores over 2000 Å diameter. For a given precursor system (viscose or phenolic), CCl<sub>4</sub> vapor sorption values correlated with BET surface area, as shown in Figure 7. All of the carbon fabrics, except for Fabric E, were extremely sorbent towards all three hazardous vapors, but no additional correlations are obvious.

The fabrics made from carbon yarns supported with other yarns, as described in Table II, have textile properties as indicated in Table IX. Fabrics L

TABLE VIII. Sorption and penetration characteristics of all carbon fabrics and yarns.

Precursor	Designation	Static CCl <sub>4</sub> Vapor Sorp., %	B.E.T. Surface Area, m <sup>2</sup> /g	Open Pore Volumes			Hazardous Vapor Exposure in 6 h (μg/cm <sup>2</sup> ) <sup>d</sup>					
				N <sub>2</sub> Pore Vol, cc/g	Hg Pore Vol, cc/g	Density g/cc	HD		GB		VX	
							Sorp.	Penet.	Sorp.	Penet.	Sorp.	Penet.
Viscose Fabric	Fabric A	17	121.6	0.05	0.91	1.39	94.6	0.4	87.5	4.8	58.1	0.0
Viscose Fabric	Fabric B	75	1210.9	0.11	1.18	1.22	95.0	0.5	89.7	0.0	49.6	0.0
Viscose Fabric	Fabric C	31	603.7	0.11	1.22	1.43	88.3	0.05	89.2	0.0	49.6	0.0
Viscose Fabric	Fabric D	45	764.0	0.04	1.25	1.30	88.3	0.05	89.1	0.05	49.6	0.0
Phenolic Fabric	Fabric E	33	947.4	0.06	1.29	1.34	94.1	0.9	56.6	35.8	28.4	28.7
Phenolic Fabric	Fabric G <sup>a</sup>	73	1342.1	0.10	1.53	1.35	100.1	0.1	—	—	—	—
Phenolic Fabric	Fabric H <sup>a</sup>	59	1127.3	0.07	1.27	1.41	—	—	93.7	0.0	—	—
Phenolic Fabric	Fabric I <sup>a</sup>	60	1385.4	0.15	1.69	1.58	—	—	—	—	51.2	0.0
Viscose Yarn	Yarn J	16 <sup>c</sup>	224.0	0.11	0.75	1.58	—	—	—	—	—	—
Viscose Yarn	Yarn K	12	154.9	0.02	1.00	1.43	—	—	—	—	—	—
None	Control <sup>b</sup>	—	—	—	—	—	98.8	1.2	40.4	59.6	97.2 <sup>e</sup>	2.8 <sup>e</sup>

<sup>a</sup> Each sample part of the same batch.

<sup>b</sup> An 8- to 9-oz/yd<sup>2</sup> cotton sateen work-clothing fabric impregnated with a chloramide and chlorinated paraffin formulation.

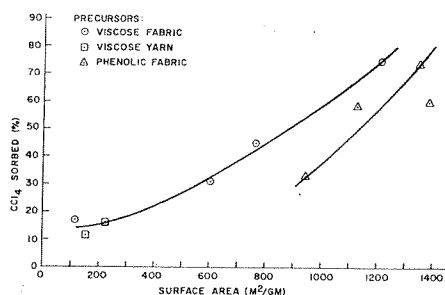
<sup>c</sup> Degassed at 3-4 mm pressure prior to test.

<sup>d</sup> Sorption + Penetration = Total Fabric Exposure.

<sup>e</sup> Test actually ran for 51 h rather than 6 h.

TABLE IX. Textile properties of fabrics constructed from carbon yarns and stronger yarns of other types.

Designation	Weight, oz/yd <sup>2</sup>		Thickness, in.	Air Perm., ft <sup>3</sup> /min/ft <sup>2</sup>	Texture, yarns/in. W×F	Breaking Strength, lb/in.	
	Total	Carbon				Warp	Fill
Fabric L	10.1	6.7	0.025	198	28 × 33	172	71
Fabric M	15.9	6.1	0.040	33	84 × 30	230	415
Fabric N	9.9	8.1	0.024	42	100 × 40	213	117
Fabric O	9.3	6.6	0.025	32	80 × 42	215	44
Fabric P	12.2	8.5	0.037	26	122 × 42	170	43

FIG. 7. CCl<sub>4</sub> vapor sorption vs. surface area for carbon fabrics and yarns.

(weft insertion warp knit), N (woven twill) and O (woven twill) had a 9 or 10-oz/yd<sup>2</sup> weight and an 0.024 or 0.025-in. thickness. The weight and thickness of Fabric M were higher due to a polyester sheath braided over the carbon yarn. Fabric P, a woven composite, was also somewhat heavier and thicker than the others. All of these fabrics sorbed significant amounts of three hazardous chemicals as indicated in Table X. Fabrics L, M, N, and P contained Carbon Yarn J and were degassed at 3–4 mm pressure prior to test to desorb contaminants acquired during storage or delivery of the carbon yarn. Fabric O contained Carbon Yarn K and was not degassed, although sorption of GB might have been greater if it had been degassed. Fabric M would have to be reconstructed with a more sorptive carbon yarn and looser braided sheath if better sorption properties are desired with HD and GB vapors.

TABLE X. Sorption and penetration data for fabrics constructed from carbon yarns and stronger yarns of other types.

Fabric Designation <sup>a</sup>	Hazardous Vapor Exposure in 6 h, µg/cm <sup>2</sup> <sup>b</sup>					
	HD		GB		VX	
	Sorp.	Penet.	Sorp.	Penet.	Sorp.	Penet.
L	99.2	0.9	73.7	19.9	49.6	1.6
M	67.1	28.4	27.9	61.2	49.6	0.0
N	92.5	3.0	83.7	5.5	49.6	0.0
O	94.1	0.9	69.9	19.8	52.8	0.0
P	100.1	0.1	92.1	1.6	51.1	0.2

<sup>a</sup> All samples except Fabric O were degassed at 3–4 mm pressure prior to test.

<sup>b</sup> Sorption + Penetration = Total Fabric Exposure.

In all of the above cases, sorption by the carbon is attributed to micropore volume filling, since no transitional pores were evident from mercury intrusion data, and only a monolayer is sorbed on the very small surface area of macropores.

Lamination of Carbon Fabric A with nylon/cotton twill and nylon tricot resulted in flexible fabric systems in the 15 to 16-oz/yd<sup>2</sup> range and with a 47 or 48 mil thickness (See Table XI). These particular systems represent weight and thickness values which are at least twice those of the original unsupported carbon fabric. As expected, lamination resulted in a decrease of air permeability and an improvement in strength. However, it did not significantly affect penetration of HD and VX vapors with respect to the control fabric. In the case of GB vapors, it is not understood why the reinforced systems showed a slight increase in penetration levels. The type of adhesive fabric used had

TABLE XI. Properties of carbon fabric laminated between nylon/cotton twill and nylon tricot.

Identification	Adhesive	Break Str., lb/in.		Air Perm., ft <sup>3</sup> /min/ft <sup>2</sup>	Thickness, in.	Weight, oz/yd <sup>2</sup>	Penetration of Hazardous Vapors in 6 h, µg/cm <sup>2</sup>		
		W	F				HD	GB	VX
Laminate Q	Polyamide	155	65	24	0.048	16.4	0.7	13.0	0.0
Laminate R	Polyester	158	56	32	0.048	15.7	0.5	8.6	0.0
Laminate S	Polyolefin	144	68	30	0.047	15.0	0.7	8.3	0.0
Carbon Fabric A (control)	Not laminated	7	14	178	0.021	6.6	0.4	4.8	0.0

little effect on overall characteristics; however, the polyolefin type gave the lightest weight system.

Quilt T, produced by quilting Carbon Fabric E between two layers of Nomex fabric, gave textile test results similar to those for the laminates (See Table XII). Reinforcement was accomplished with trade-off on reduced air permeability and increased thickness and weight. However, in the sorption tests it was not understood why quilting produced higher penetration levels from HD and GB vapors and a lower penetration level for VX.

It is desirable to have sufficient characterization of the carbon to explain why some all-carbon fabrics and yarns sorb more vapors than others. Accordingly, small pore volume analyses were made (Table XIII) and molecular diameters of various vapors (Table XIV) were calculated. Diameters were obtained from data on molecular weight, density at 20 to 25°C, and an assumption of closely packed spherical molecules by one method and estimated from ranges of dimensions for various configurations of Fisher-Hirschfelder-Taylor Atom Models (Fisher Scientific Co. kits) in a second method.

It was found that all pores from 7 Å diameter up should be available to the vapors selected. However, no definite pattern of micropore volume filling by the different molecular diameter vapors was discernable to explain sorptivity differences in the materials.

The experimental sorption plots in Figure 8 elucidate the potential of carbon fabric for liquid phase sorption. These Freundlich sorption isotherms are for two dyes,

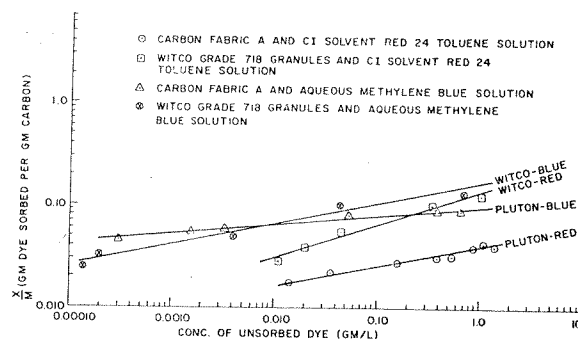


Fig. 8. Freundlich sorption isotherms for dye solutions with activated carbon fabric and granules.

TABLE XII. Quilting of carbon fabric between two layers of nomex fabric.

Identification	Break Str., lbs/in.		Air Perm., ft <sup>3</sup> /min/ft <sup>2</sup>	Thickness, in.	Weight, oz/yd <sup>2</sup>	Penetration of Hazardous Vapors in 6 h, µg/cm <sup>2</sup>		
	W	F				HD	GB	VX
Quilt T (Carbon Fabric E, quilted)	325	305	7	0.035	11.5	8.1	55.2	7.0
Carbon Fabric E (control)	5	7	151	0.020	5.3	0.9	35.8	28.7

TABLE XIII. Analysis of small open pore volumes from nitrogen sorption isotherms of all-carbon fabrics and yarns.

Carbon Material	Volume (cc/g) for Various Pore Diameter Ranges											
	7-10Å	10-20Å	20-30Å	30-40Å	40-50Å	50-60Å	60-70Å	70-80Å	80-90Å	90-100Å	100-200Å	200-300Å
Fabric A	0.0155	0.0302	0.0033	0.0003	0.0000	0.0000	0.0000	0.0001	0.0000	0.0000	0.0001	0.0006
Fabric B	0.0362	0.0476	0.0112	0.0046	0.0030	0.0010	0.0010	0.0008	0.0007	0.0004	0.0025	0.0009
Fabric C	0.0233	0.0289	0.0035	0.0013	0.0005	0.0003	0.0002	0.0002	0.0001	0.0001	0.0010	0.0004
Fabric D	0.0147	0.0210	0.0025	0.0007	0.0004	0.0003	0.0002	0.0001	0.0001	0.0001	0.0010	0.0007
Fabric E	0.0334	0.0458	0.0055	0.0029	0.0020	0.0010	0.0008	0.0006	0.0003	0.0002	0.0013	0.0003
Fabric G	0.0447	0.0439	0.0054	0.0018	0.0008	0.0006	0.0004	0.0002	0.0002	0.0002	0.0012	0.0005
Fabric H	0.0316	0.0316	0.0036	0.0008	0.0004	0.0004	0.0003	0.0001	0.0000	0.0001	0.0008	0.0002
Fabric I	0.0651	0.0668	0.0090	0.0030	0.0016	0.0009	0.0003	0.0004	0.0004	0.0003	0.0014	0.0004
Yarn J	0.0168	0.0563	0.0226	0.0063	0.0029	0.0014	0.0004	0.0002	0.0004	0.0004	0.0017	0.0006
Yarn K	0.0092	0.0074	0.0009	0.0002	0.0002	0.0001	0.0001	0.0001	0.0001	0.0001	0.0008	0.0006

TABLE XIV. Molecular diameters of vapors sorbed.

Molecule	Spherical Diameter From Density, Å	Model Dimensions, Å				
		Length	Ranges Width	Height	Thinnest Model Width	Height
CCl <sub>4</sub>	6.7	6	6	6	6	6
HD	7.3	8-12	5-6	4-6	5	5
GB	7.4	8-10	7	5	7	5
VX	9.4	11-17	7-11	6-8	7	8



each sorbed from a different solvent onto both the fabric and granular forms of carbon. The difference in slopes and a crossover point were prominent features of the isotherms of aqueous Methylene Blue with fabric and granules. The fabric appeared to be a more effective sorbent for this dye at lower concentrations and maintained its effectiveness at higher concentrations [4]. Conversely, the granules were consistently more sorbent than the fabric with respect to sorption of CI Solvent Red 24 from toluene. This liquid phase sorption is primarily relevant to chemical processing, but it is also a factor to be considered in protective clothing, insofar as sorption of sweat (aqueous solutions) can reduce the sorption sites of active carbon available for hazardous chemicals.

Data relevant to clothing comfort are summarized in Table XV. The first four samples, each representative of a different carbon fabric system, indicated that relatively low thermal insulation in the 1.32 to 1.45 clo<sup>7</sup> range is obtainable with a wide variation of weight, thickness, and air permeability. The fifth sample, a quilt notable for its low air permeability, had a high clo value of 1.77, thus indicating it would be the most uncomfortable of the series in hot climates. For comparison purposes this table shows the properties of a normal business suit fabric which is lighter in weight, thinner, and has a lower thermal insulation value.

### Conclusions

Surface area measurements and carbon tetrachloride sorption data indicated that all-carbon fabrics prepared by Arons and Macnair (phenolic precursor) and Maggs (viscose precursor) were the most activated forms of all-carbon textiles studied. It is probable that the lower-activity carbon yarns studied can be improved to have comparable high sorption capacity. The all-

<sup>7</sup> The unit "clo" indicates the insulation value of clothing, where one clo is required by an individual at rest for comfort at normal room temperature. One clo represents a clothing conductance value of 1.14 Btu/ft<sup>2</sup>h °F (5.55 Kcal/m<sup>2</sup>h °C).

carbon fabrics were quite weak, but the carbon yarns had minimal strength with a tenacity comparable to that of a typical cotton yarn.

In general it has been shown that activated carbon fibers in the form of fabric or yarn can effectively sorb gases, vapors, and dissolved solids. These carbon forms can be designed to acquire considerable mechanical reinforcement by incorporation into permeable textile systems. Reinforcement is obtained, for example, by laminating or quilting all-carbon fabric between two other fabric layers or by knitting or weaving carbon yarns with other yarns. However, reinforcement of the all-carbon fabrics is accomplished at the expense of increased weight and discomfort in hot climates.

Considerable experimentation is needed to determine the ranges of physical and chemical properties attainable for activated carbon fabrics, yarns, and their reinforced systems. The reinforced systems appear to have potential for use in protective clothing applications, but thinner and lighter weight materials are desirable for hot climates.

ACKNOWLEDGMENT. Mrs. Barbara Kirkwood, U. S. Army Natick Laboratories, cooperated in this effort by performing thermal insulation tests.

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TABLE XV. Clothing comfort factors of fabrics containing carbon fibers.

Identification	Weight, oz/yd <sup>2</sup>	Thickness, in.	Air Perm., ft <sup>3</sup> /min/ft <sup>2</sup>	Thermal Insulation, <sup>a</sup> clo
Fabric L (Knitted carbon and Nomex yarns)	10.1	0.025	198	1.37
Fabric N (Woven carbon and Nomex yarns)	9.9	0.024	42	1.32
Fabric P (Composite woven with carbon, Nomex, and cotton yarns)	12.2	0.037	26	1.41
Carbon Fabric (Laminate S)	15.0	0.047	30	1.45
Carbon Fabric (Quilt T)	11.5	0.035	7	1.77
Business Suit Fabric	8.0	0.010 to 0.015	20 to 40	1.0

<sup>a</sup> Includes 0.75 clo for dead air layer on body surface.

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Manuscript received February 7, 1974.